

DYNAMIC MECHANICAL ANALYSIS OF POLYMERIC MATERIALS

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ABSTRACT

Polymeric materials exhibit mechanical behavior which is dependent on temperature. Dynamic mechanical analysis measures the mechanical damping and resonant frequency of a material over a temperature range. Values of the dynamic loss modulus, storage modulus, and loss tangent can be calculated from these data. The glass transition temperature and onset temperature are obtained from curves of the dynamic moduli versus temperature.

INTRODUCTION

Polymeric materials do not deform easily at low temperatures. This is referred to as the glassy state. At high temperatures, the same material will be rubbery and will deform easily. The temperature at which this change in behavior occurs is called the glass transition temperature, T_g . The transition usually occurs over a temperature range, called the glass transition region. Materials are structurally sound at temperatures below the onset to the glass transition region, hence the importance of determining the glass transition temperature and the onset temperature.

The response, or strain, of a polymeric material to a sinusoidal stress is characterized by a modulus. The modulus, the ratio of stress to strain, is composed of the storage modulus and the loss modulus. The storage modulus is related to the amount of energy stored in the material as a deformation and returned to the oscillation, while the loss modulus is related to the amount of energy lost through friction.

When the temperature is low (glassy state), the loss modulus is small and the storage modulus is large. As the temperature increases, the intermolecular friction changes and the loss modulus increases (transition region) to a maximum (T_g). At still higher temperatures, the loss modulus decreases (rubbery state), and the storage modulus is also small.

Dynamic mechanical analysis of polymeric materials is analogous to the simplified, linear model shown in Figure 1. The equation of motion for this damped, driven harmonic oscillator is

$$M \frac{d^2x}{dt^2} + B \frac{dx}{dt} + Kx = F_0 \sin \omega t$$

where M is the mass of the oscillating body, B is the coefficient of friction, K is the spring constant, x is the displacement of the mass from its equilibrium position, and F_0 is the magnitude of the applied sinusoidal force. For oscillation at resonant frequency, the position of the mass is 90° out of phase with the applied force, and can be written

$$x = x_0 \cos \omega t$$

where x_0 is the maximum displacement. Substituting this expression with its first and second derivatives into the differential equation of motion and comparing the coefficients of the sine and cosine terms, we obtain the equalities

$$K = M\omega^2$$

and

$$\omega B = -F_0/x_0$$

The first of these equations shows that the spring constant, which represents the ability of the oscillation mechanism to store energy, is proportional to the square of the resonant frequency. This is analogous to the storage modulus of a material. The second equation shows that the damping term, representing the loss of energy from the system, is proportional to the applied force and inversely proportional to the magnitude of the oscillation. This is analogous to the loss modulus of a material.

EXPERIMENTAL

The DuPont¹ 982 Dynamic Mechanical Analyzer (DMA) is used to measure the mechanical damping and resonant frequency of a sample of material as a function of sample temperature. The sample is clamped between the ends of two rigid arms, each of which is free to oscillate around a pivot point (See Figure 2). An electromagnetic driving mechanism is connected to the opposite end of one arm. The driving frequency is varied by the apparatus until the driving moment, sensed from the driving current, is a minimum as a function of frequency, provided the amplitude is fixed. This frequency is the resonant frequency and the apparatus adjusts to the resonant frequency which changes with temperature. The oscillation amplitude is monitored by a linear variable differential transformer, which provides a feedback signal to the driving mechanism to maintain a constant amplitude. This same feedback signal, in millivolts, is recorded as a measure of energy lost during each cycle or the damping due to the specimen. With the use of liquid nitrogen, the system can record the resonant frequency and damping of a material from -150°C to 500°C . See Figure 3.

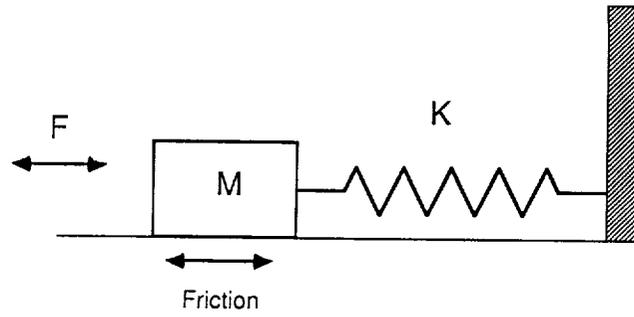
The 982 DMA is accompanied by the 1090 Thermal Analyzer (TA), shown in Figure 4, which is used for data acquisition, data analysis, and temperature control. Data analysis programs provided by DuPont calculate the loss and storage moduli from damping and frequency values stored by the 1090 TA. The frequency and damping signals of the arms with no sample and with a high modulus, low loss material, such as steel, are used to calibrate the instrument.

ANALYSIS

An example of the resulting curves for a sample of T300/934 graphite/epoxy composite is shown in Figure 5. The transition onset temperature is found by drawing tangents to the loss modulus curve before the transition region and during the increasing part inside the transition region, as shown in Figure 5. The intersection of these two tangents is considered to occur at the onset temperature. The glass transition temperature is taken to be the temperature at which the loss modulus reaches a peak.

CONCLUSION

The data shows that the glass transition temperature is 240°C ($\pm 5^{\circ}\text{C}$) for T300/934 graphite/epoxy composite. The onset temperature is 218°C ($\pm 5^{\circ}\text{C}$), below which the material would be structurally sound.



$$M \frac{d^2x}{dt^2} + B \frac{dx}{dt} + Kx = F_0 \sin \omega t$$

For oscillation at resonant frequency,

$$x = x_0 \cos \omega t$$

$$K = M\omega^2$$

$$\omega B = -F_0/x_0$$

Figure 1. The simplified linear model of dynamic mechanical analysis. A mass M attached to a spring of elasticity, K , is driven by an applied sinusoidal force, F . Energy is lost from the oscillation by friction.

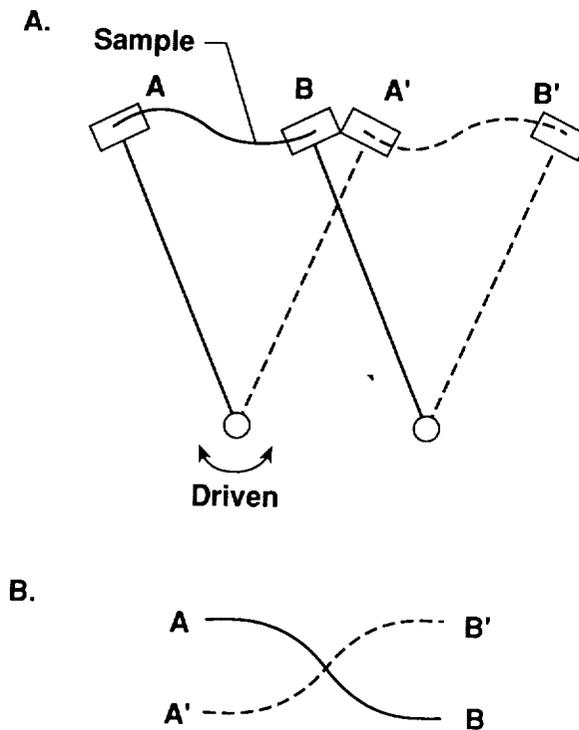


Figure 2. The dynamic mechanical analyzer. A. The oscillation mechanism consists of two arms with a driving mechanism. The sample is clamped at the ends of the arms. B. The sample is flexed in the manner shown (exaggerated here).

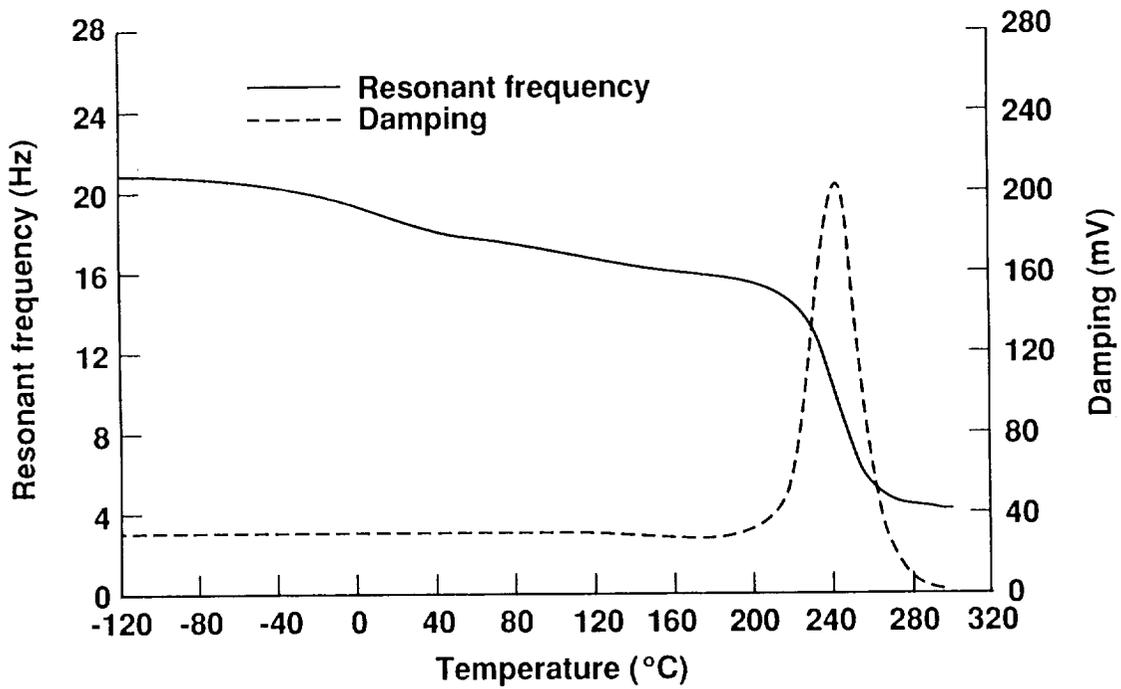


Figure 3. Frequency and damping signal over temperature for a sample of T300/934 graphite/epoxy composite. The resonant frequency decreases in the glass transition region as the stiffness of the sample decreases.

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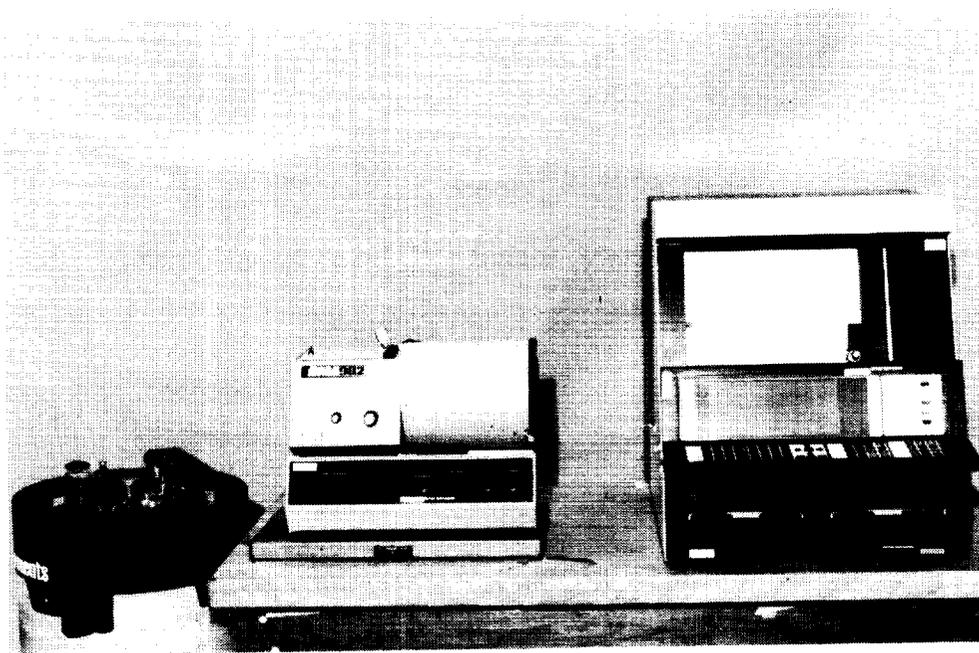


Figure 4. The DuPont 1090 Thermal Analyzer, right, and 982 DMA with heating chamber in place. At the left is the liquid nitrogen tank.

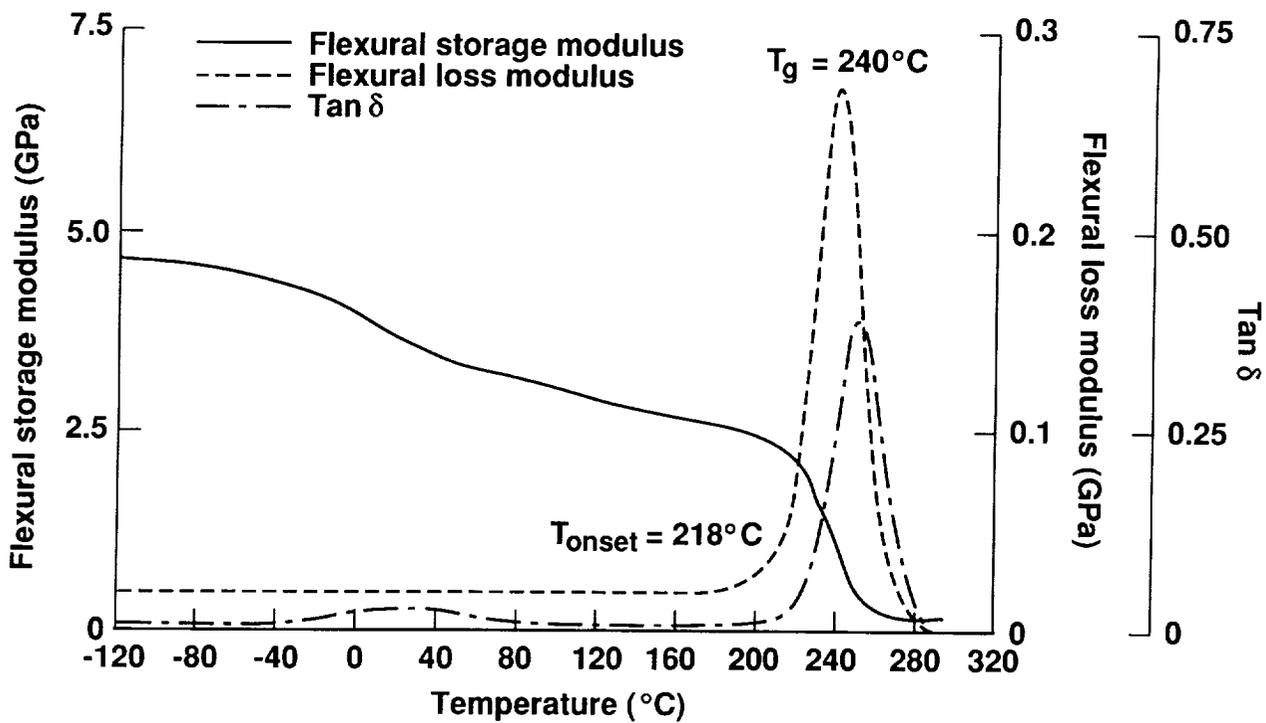


Figure 5. Plots of the dynamic moduli and loss tangent versus temperature calculated from the data on a sample of T300/934 graphite/epoxy composite.